

Microstructures and properties of superconducting Y-ErBaCuO thin films obtained from disordered Y-ErBaF₂Cu films

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1. INTRODUCTION

High T_c Superconducting thin films can be prepared either by an ex-situ or an in-situ process. Although the latter route is more appropriate in applications that require samples of small areas (or preparation of superlattices) the former still maintain a certain appeal for those applications in which a single layer of films of large area are needed, e.g., coating of radiofrequency cavities. Moreover the use of BaF₂ in the preparation of the precursor films, used in the ex-situ preparation process, makes these films much more moisture-resistant. Here we describe in all the details the preparation procedure used to obtain superconducting thin films by R.F. magnetron sputtering of a single mosaic target composed by (Y-Er), BaF₂ and Cu. There have been several reports¹⁻¹⁵ on the production of high T_c superconducting thin films obtained from precursor films composed by Y, BaF₂, and Cu, but to our knowledge this is the first case in which such films are obtained simply by means of sputtering of a single mosaic target^{12,13}.

2. SPUTTERING CONDITIONS AND PRECURSOR FILMS

The R.F. magnetron target has been made by sticking small pieces of Yttrium-Erbium and Barium Fluoride on a Copper disk whose diameter is 200 mm. Standard R.F. discharge conditions are: 5×10^{-3} Torr, 400 Watt, pure Argon gas. The precursor films obtained have the expected 1:2:3 composition on a diameter of about 100 mm. Typical X-ray spectra of the precursor films are shown in fig. 1. Oxygen passivation is needed in order to obtain films with a smooth surface. The X-ray spectra of a precursor film with a smooth surface (curve A) show only the 111 line characteristic of the BaF₂ cubic structure. X-ray spectra of

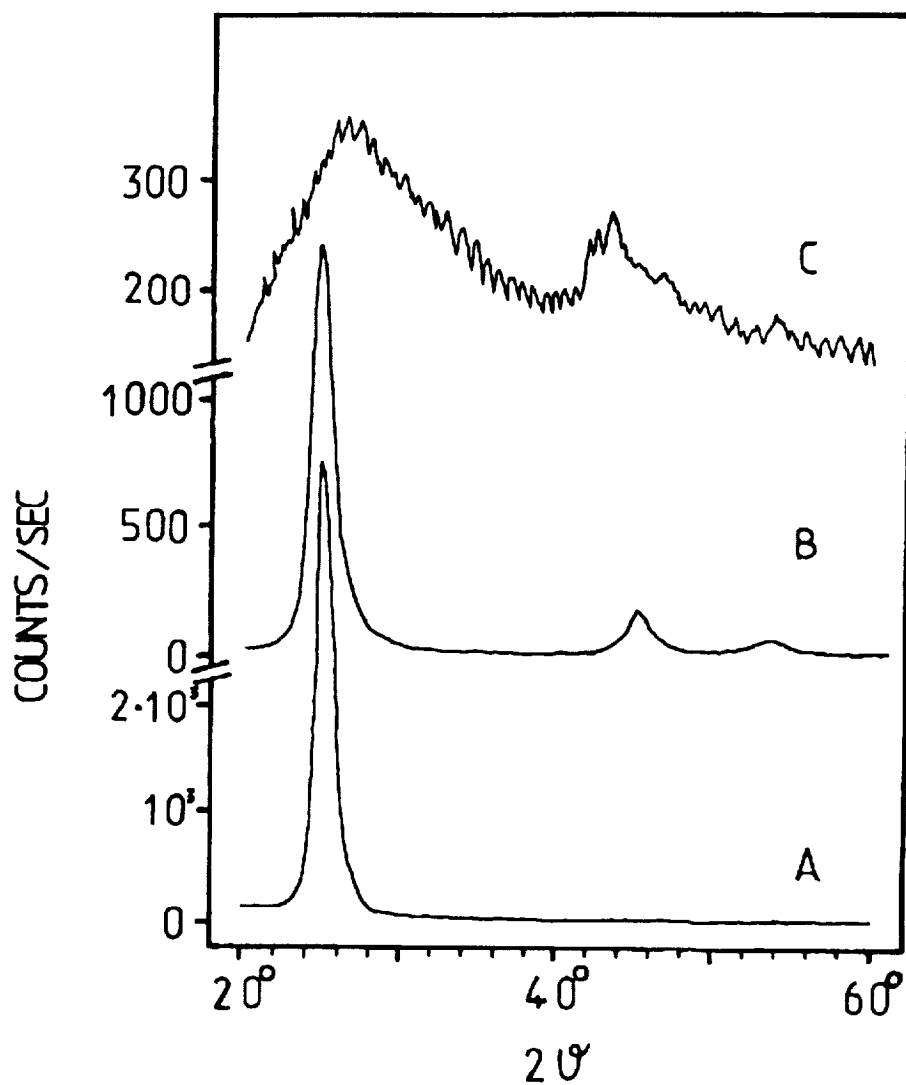


Fig. 1 - X-ray spectra of precursor films: A - passivated sample, B - unpassivated sample, C - unpassivated sample after two month.

unpassivated samples evidenciate a much less intense 111 BaF_2 line and bumps characteristic of the Cu f.c.c. structure indicating a partial copper segregation.

3. ANNEALING PROCEDURE

A typical annealing profile is shown in fig. 2: note the use of water in order to remove the Fluorine from the sample. The pictures of fig. 3 show how the temperature of the higher plateau affects the formation of the crystallites in the case of a sample 7500 Å thick. In fig. 3a

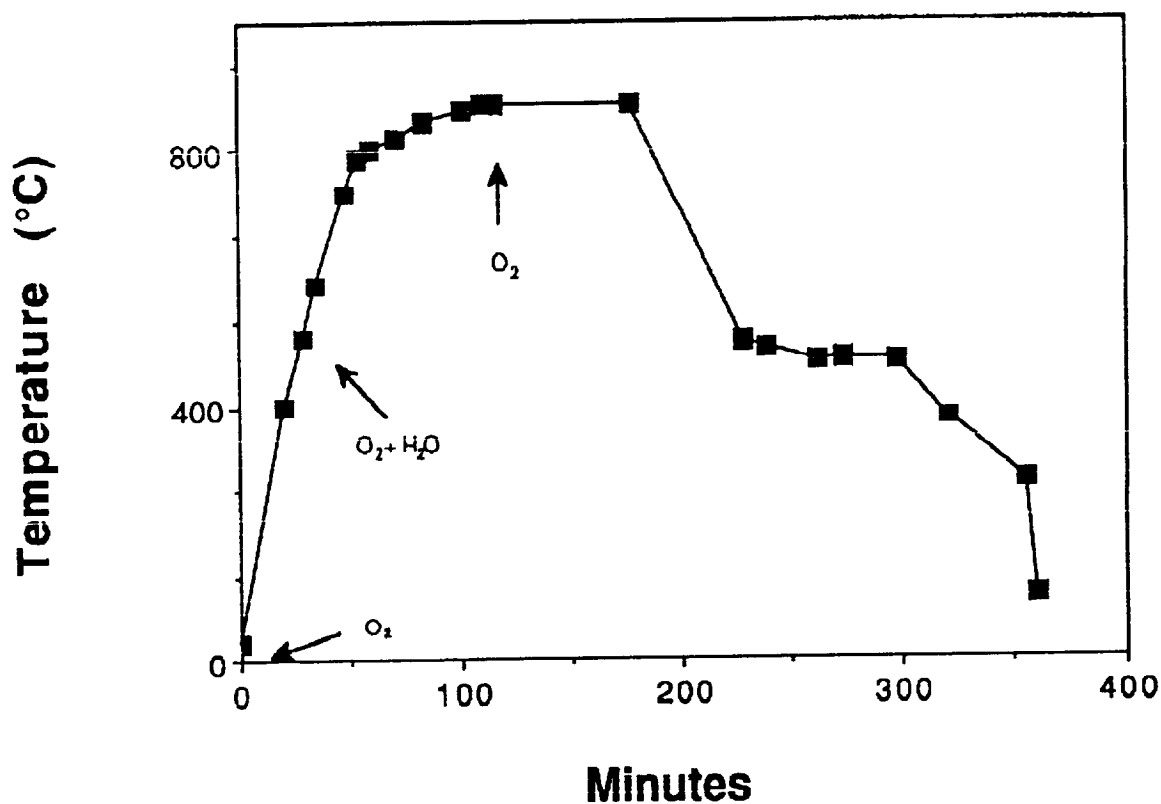
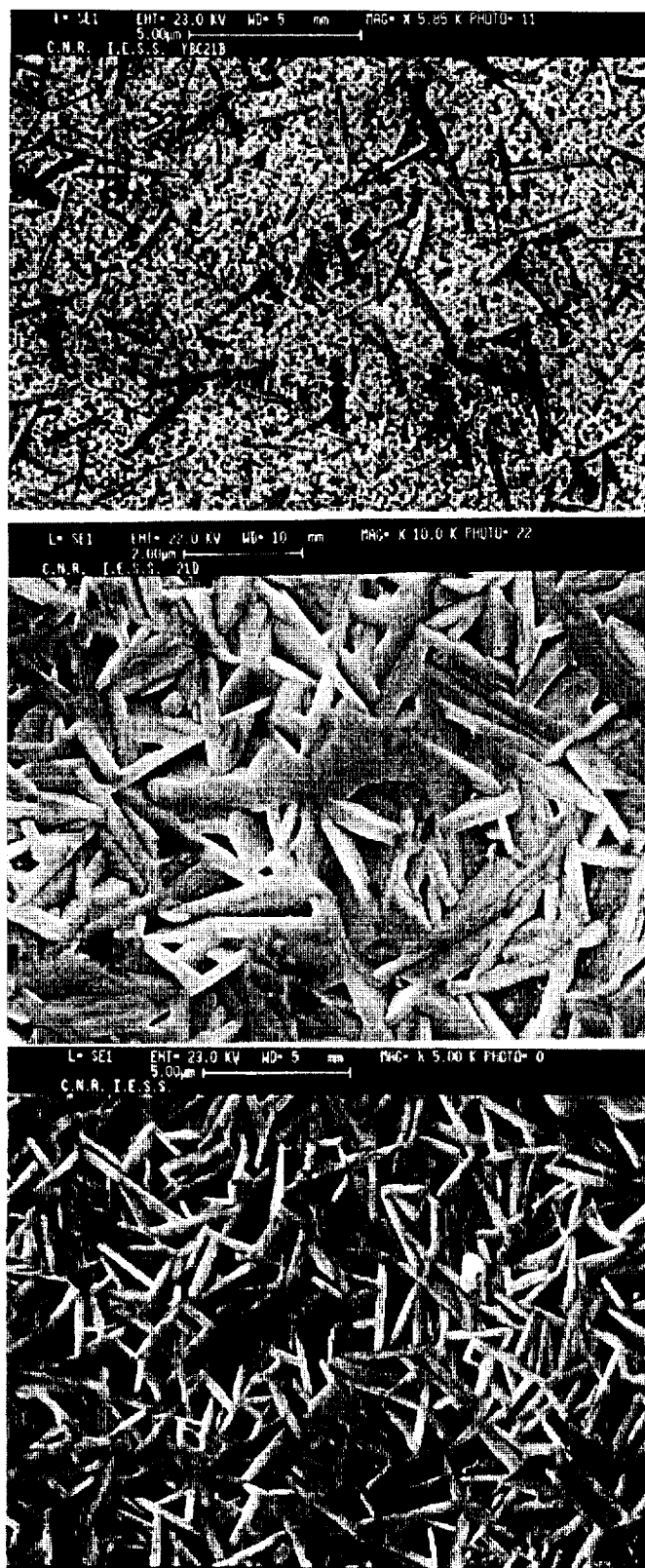


Fig. 2 - Typical annealing profile.

we observe a certain amount of crystallites that, we believe, enucleate either from the surface of the substrate and from the bulk of the film; the black spots also visible are superconducting areas from which the formation of the crystallites develops.

Fig.s 3b and 3c correspond to top-plateau temperatures of 830 and 850 °C respectively; in the latter we observe fully developed crystallites. They are randomly oriented and from their size we can deduce that the growth rate in the direction of the a-b plane is at least a factor 10 higher than in the c direction. In correspondence with the increasing degree of crystallinity of

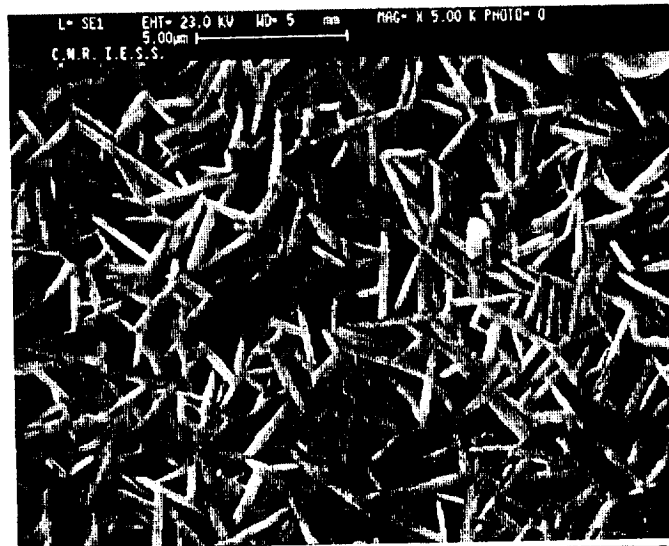


a

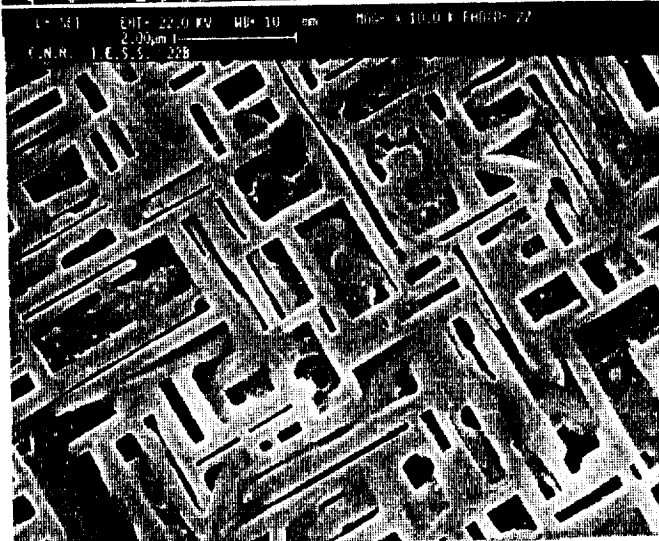
b

c

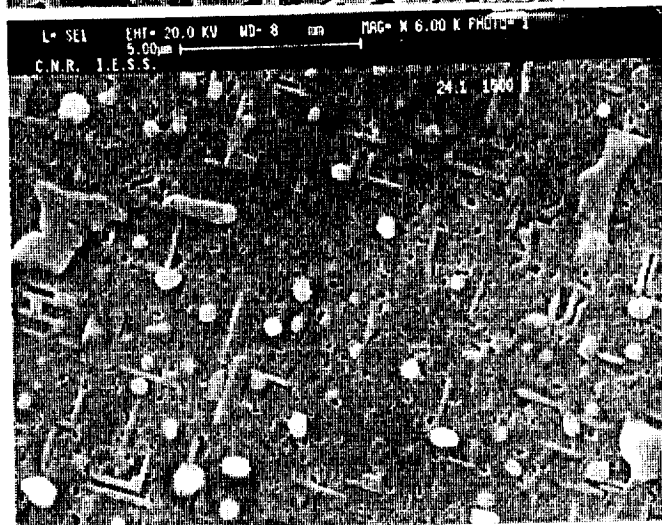
Fig. 3 - SEM pictures of samples , all having the same thickness (7500 Å) but that have been subjected to annealing cycles having a different temperature of the top plateau (see fig. 2): a - 820 °C, b - 830 °C, c - 850 °C. Note the different scale used for sample b.



a



b



c

Fig. 4: SEM pictures of samples having different thickness
a: 7500 Å, b: 3000 Å, c: 1500 Å.

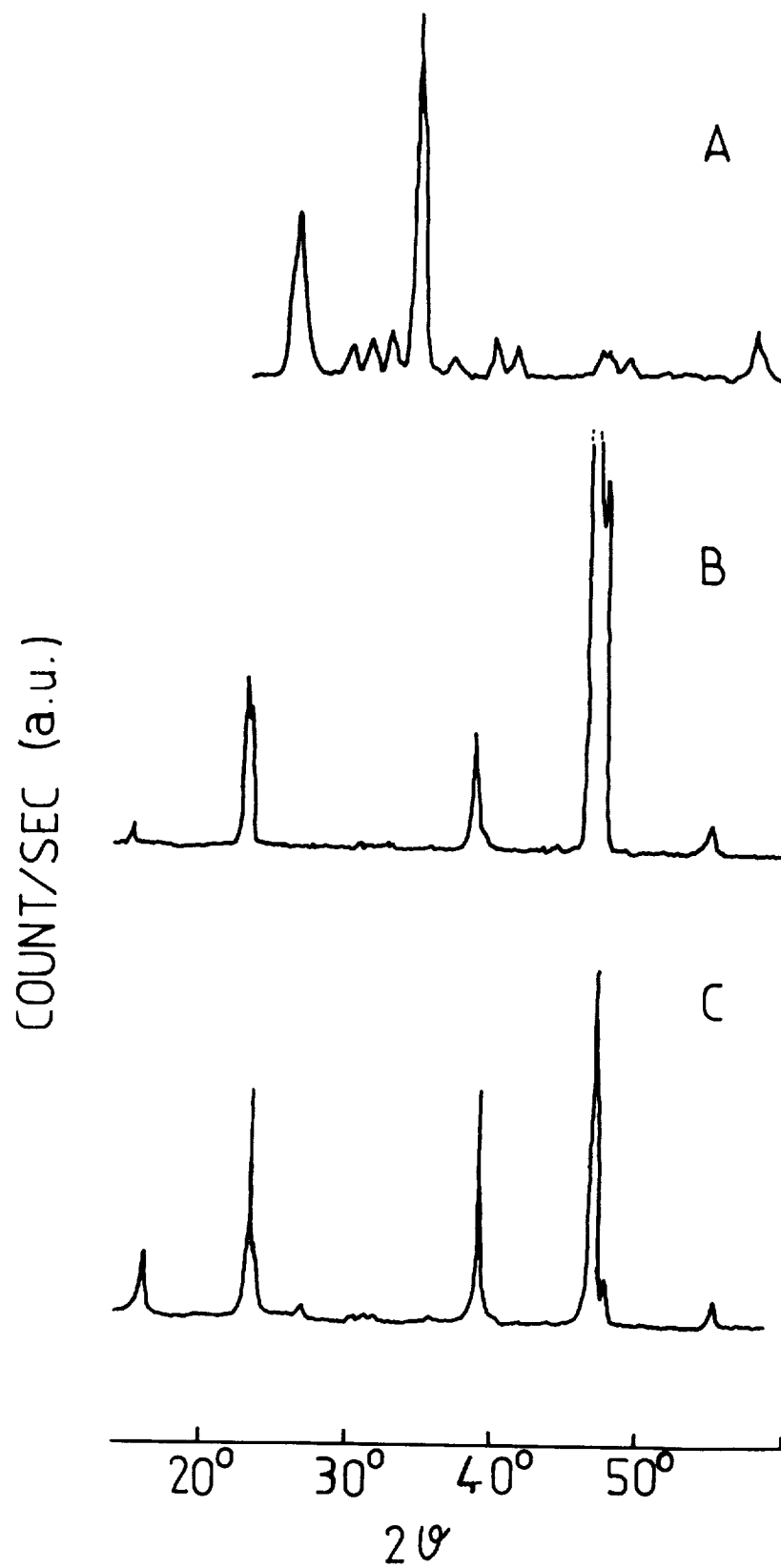


Fig. 5: X-ray data of thin films whose SEM pictures are shown in fig. 4

the film we observed in the x-ray spectra, as expected, a decrease down to zero of the intensity of the 111 BaF₂ line.

4. EFFECT OF SAMPLE THICKNESS

In fig. 4 we show SEM pictures of samples having different thicknesses, but submitted to the same annealing conditions (fig. 2). The corresponding X-ray data are reported in fig. 5. Sample a is clearly composed by random crystallites. To a first glance the X-ray spectra corresponding to samples b and c suggest the presence of a c-texture. However the SEM-picture of sample b clearly shows a cross-linked structure typical of crystallites having the c-axis lying along the plane of the substrate and the a-axis perpendicular to it;¹⁰ their dimensions confirms the slower growth rate along the c-axis of factor between 5 and

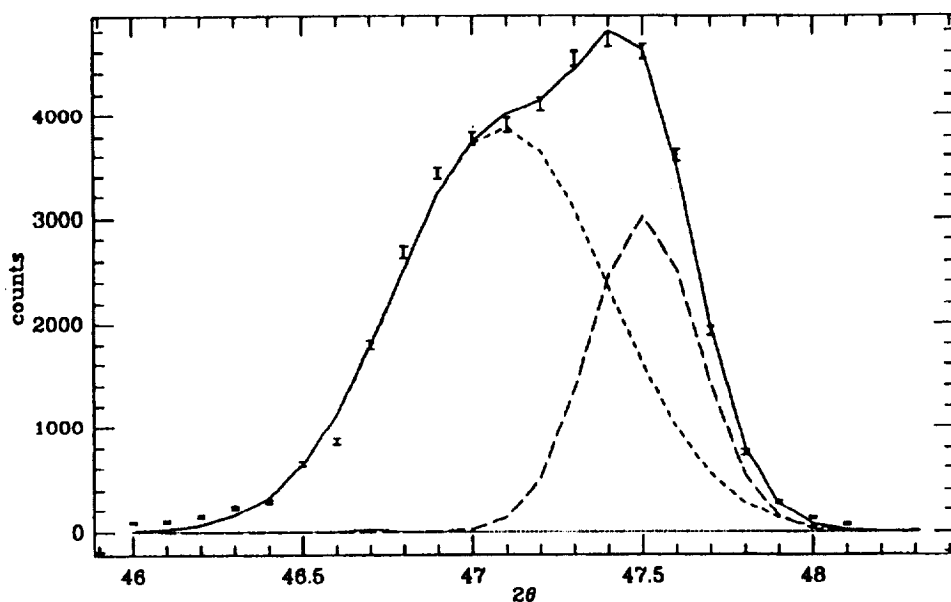


Fig. 6 - Three-gaussian fit to the x-ray line-shape for the sample in fig.4b; the two peaks around $2\theta = 47^\circ$ and $2\theta = 47.5^\circ$ are the (200) and (006) lines respectively. The third peak, around $2\theta = 46.7^\circ$, barely visible on the scale of the plot, is the (200) line from the substrate.

20.^{16,17} An accurate analysis of the line-shape observed around $2\theta = 47^\circ$ (see fig. 6), shows indeed the existence of both textures. In sample c a-textured crystallites that nucleate

directly from the substrate are rare although not completely absent, and the sample results to be almost fully c-textured. The fact that in the thinner sample we observe basically only c-oriented crystallites leads us to suggest that sample b is constituted by a c-textured layer grown epitaxially on the substrate and by an a-textured layer heteroepitaxially grown on the top of the c-textured one. The onset temperature of the resistive transition for these three samples are 86.8 K, 86 K, and 80.5 K respectively. The sharpness of the resistive transition as well as the onset temperature are, basically, correlated with the quality of the surface layer (i.e. that visible in the SEM pictures) but not necessarily with the X-ray spectra. Indeed we observed also cases in which the X-ray spectra show sharp 00n peaks, in contrast with the SEM picture showing a random surface with poorly formed crystallites; furthermore, the onset temperatures in such cases are lower than 77 K.

5. CONCLUSIONS

We have shown that a combination of several techniques is necessary to characterize properly each step of the preparation of the high T_c superconducting thin films. C-textured films are easily obtained in the thickness range 1000 - 2000 Å while thicker films are constituted by a heteroepitaxial a-textured layer grown on the top of a c-textured one. This is an extremely important observation because it might explain several discrepancies reported in the measurements of physical quantities obtained from nominally (but not fully characterized) c- or a-textured samples. For thicknesses higher than 4000 - 5000 Å the samples are randomly oriented.

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